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Utilization of Guinea corn (*Sughurm vulgare*) Husk for Preparation of Bio-based Silica and it's Characterization Studies

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Abstract*—Bio-based silica was prepared from Guinea corn Husk within the context of sustainable chemistry. The Guinea corn Husk was leached in dilute HCl, washed, dried and calcined at 650 ºC for 4hr. The resultant ash was digested in 2M NaOH solution, precipitated by adding H_2SO_4 and then washed to remove sulphate by-product. The siliceous materials were characterized using SEM-EDX, FTIR and XRD to investigate its composition and morphology, functional groups and the phase respectively. The elemental investigation revealed Si as major element in the ash. The presence of Si-O-Si (siloxane) and Si-OH (silanol) were confirmed by FTIR of the silica. The SEM micrograph of the silica also showed agglomeration of regular spherical particles in the morphology. The XRD pattern of the siliceous material indicated an amorphous form of the product. Guinea corn Husk as an agricultural waste can be a sustainable means for silica and other siliceous materials.*

Keywords*— *Agricultural waste, Bio-based, Guinea corn Husk, Silica, Sustainable chemistry.

I. INTRODUCTION

The concerns of sustainable chemistry have lead to the production of bio-based materials. Bio-based materials refer to products that mainly consist of a substance (or substances) derived from biomass and either occur naturally or are synthesized, or it may refer to products made by processes that use biomass (Curran, 2010). Sustainable chemistry has one of its objectives to be the use of renewable feed stocks and byproduct in the production processes (Karagolge and Gur, 2016). Sustainable means are used to find alternatives to conventional chemical syntheses and transformations (Varma, 2014). Scalet *et al.*, (2015) posited that this sustainable means play a significant role in effectively addressing the global challenges, such as economy, climate change, limited natural resources, dependency on decreasing fossil resources.

Agriculture practice is a means of sustainable development as it contributes majorly to the economy of some countries in the world (Chongela, 2015). Recently more attention is given to agricultural production in most developing countries. In Nigeria, part of the efforts of the government to diversify economy of the nation from non-renewable oil is by boosting agricultural production (Anyawu *et al.*, 2013). Waste from agricultural practices have been investigated and reported to have a greater potential for bioresource like bioenergy (Simonyan and Fasina, 2013) and biogas (Ngumah *et al.*, 2013).

Guinea corn Husk (GcH) is an agricultural waste from milling of Guinea corn (*Sughurm vulgare*). Guinea corn is an important food crop grown abundantly in northern part of Niger and Benue Rivers in Nigeria (Ndububa and Nurudeen, 2015). Akinloye *et al.* (2014) reported that about 1.5 million tones of guinea corn husk is generated annually which has potential to increase as the economy is diversified to agriculture. Guinea corn Husk has been utilized in the production of bioethanol (Oyeleke and Jibrin, 2009) and its ash as partial replacement in Ordinary Portland Cement in concrete (Ndububa and Nurudeen, 2015) with major constituent of SiO_2.

Silica, SiO_2 is present in abundance on earth crust and plant root tissues (Ghorbani *et al.,* 2013). It exists in amorphous and crystalline structures consisting of inter-linked SiO_4 unit in a tetrahedral arrangement (Nandanwar, 2013; Le *et al*., 2013). It is an important basic raw material in many industrial finished products such as electronics, ceramic, pharmaceutics, detergents, adhesives and polymer materials. It also has many technological applications as thixotropic agents, thermal insulators, composite fillers, e.t.c. (Ghorbani *et al.,* 2013; Sun and Gong, 2001). Some biomass materials have been reportedly used for synthesis

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of silica among which are rice husks (Wang, 2012; Le *et al.,* 2014), wheat husks (Shaik and Shaik 2013), coconut shells (Sivasubramanian and Sravanthi, 2015), sedge (Ghorbani *et al.,* 2013), cow dungs (Rani, *et al*., 2014).

Consequence of the expansion in agricultural production is an increase in the level of agricultural waste. These agricultural wastes encompass waste from unutilized (excess) and biomass residue from processing of agricultural products (Edewor-Ikuponiyi and Amuda, 2013). Often, these wastes pose threat to the environment, resulting in bad odour and also affecting the quality of groundwater. However, agricultural wastes embedded with various useful constituent which can be harnessed as raw materials for further production of materials.

Attention of researchers is now shifted to the utilization of biomass material towards a sustainable development of chemicals. Therefore, the focus of this research is on the possibility of preparing siliceous materials from Guinea corn Hush.

II. MATERIALS AND METHODS

2.1 Materials

Guinea corn Husk (GcH) was sourced locally from farmer as an agricultural waste and was used as a feedstock for the preparation of silica. Chemical reagents used including sodium hydroxide, sulphuric acid, hydrochloric acid and barium chloride are of analytical grade and were used as purchased.

2.2 Methods

2.2.1 Preparation of Silica from Guinea corn Husk

The preparation was done using slightly modified method reported by Sivasubramanian and Sravanthi, (2015). The guinea corn Husk (GcH) was washed thrice with de-ionized water to remove some foreign particles from it after which it was air dried. The air dried GcH was pre-treated by leaching in 0.1 M HCl for 2 hr and washed with de-ionized water to remove some other metallic oxides. The pre-treated GcH was calcined in muffle furnace at 650 ºC for 4 hr to produce Guinea corn Husk Ash (GcHA). Silica extraction was done by adding 2 M NaOH solution to the ash and stirred on magnetic stirrer plate at a constant temperature of 100 ºC for 8 hr. Afterwards, the mixture was filtered and the sodium hydroxide extract in form of sodium silicate (eqn. 1) was further used for the preparation of silica. The sodium silicate was reacted with 2.5 N H_2SO_4 to precipitate pure silica (SiO_2) (eqn. 2). The white siliceous precipitate formed was aged in an autoclave for 5 hr, filtered and washed severally with de-ionized water to remove sulphate impurities.

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O \quad (1)$$

$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O \quad (2)$$

2.2.3 Characterization Studies

The elemental compositions of the guinea corn husk ash (GcHA) were determined by X-Ray Fluorescence (XRF) and Energy Dispersive X-ray Spectroscopy (EDX). The bio-based silica prepared from the GcHA was characterized for its surface functional groups, morphological properties with elemental compositions and to assess the phase, the degree of crystallinity using FTIR and SEM-EDX and XRD respectively.

III. RESULTS AND DISCUSSIONS

Guinea corn Husk Ash and Silica

The composition of the acid-leached Guinea corn Husk Ash (GcHA) determined by XRF is presented in Table 1. The results showed that the ash contains 93.83% of SiO_2 as the major component; Al_2O_3 and CaO were present as minor components while K_2O, Fe_2O_3 and P_2O_5 as trace components. This indicates that guinea corn husk (GcH) is a good precursor for silica comparing with value reported for rice husk ash (99.08%) by Le *et al.,* (2013). In a related investigation on the same guinea corn husk by Nurudeen and Ndububa, (2015), 78.17% of SiO_2 was reported. However, 93.83% obtained from this study is likely due to the pre-treatment in HCl which reduced the content of other metallic oxides.

Table.1: Chemical composition of the GcHA

Element	**Al_2O_3**	**SiO_2**	**K_2O**	**CaO**	**Fe_2O_3**	**P_2O_5**
% Weight	1.182	93.83	0.069	1.897	0.146	0.247

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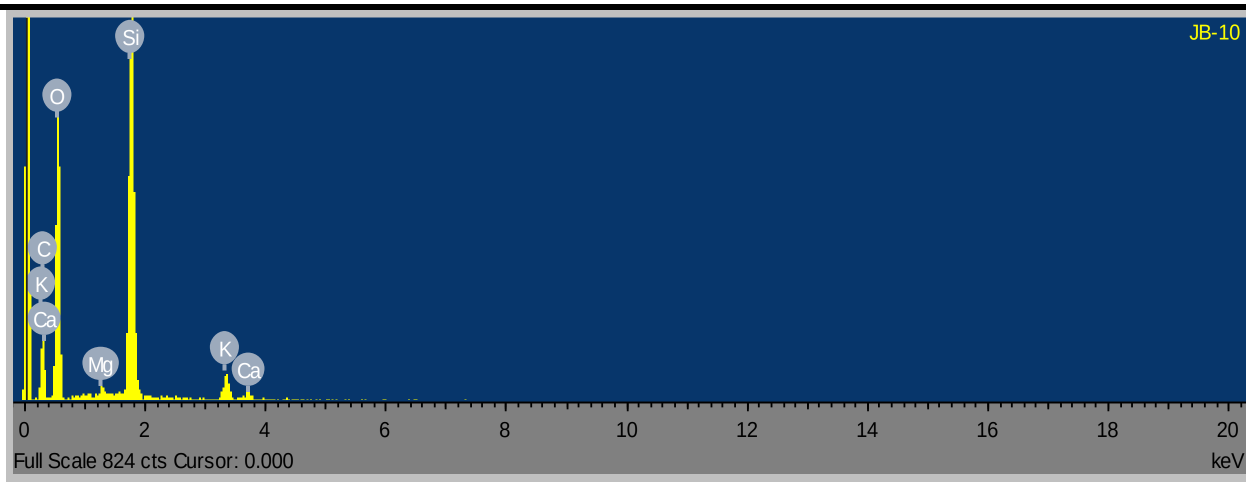

Fig.1: EDX spectrum of Guinea corn Husk Ash (GcHA)

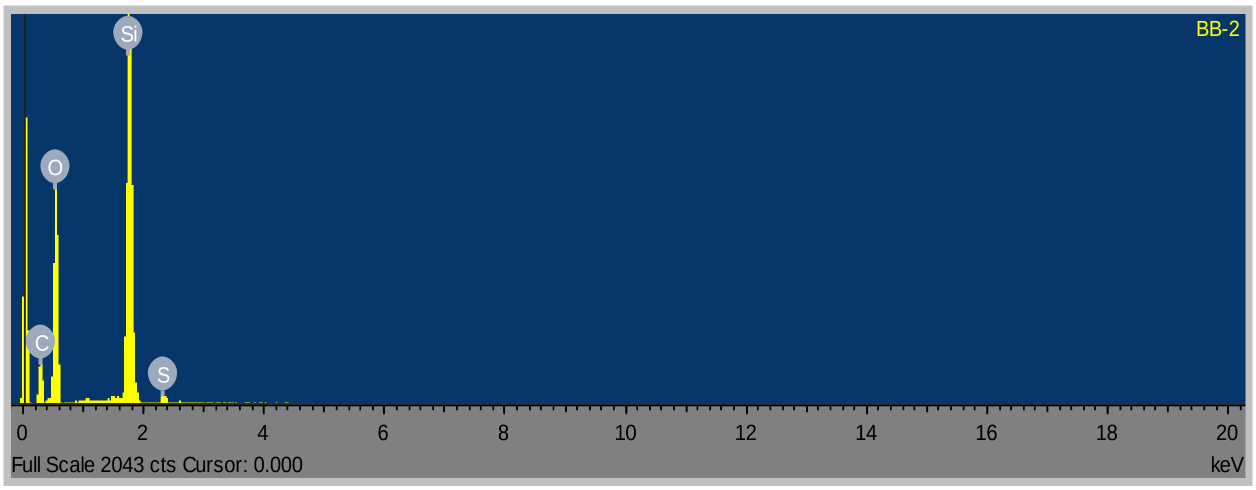

Fig.2: EDX spectrum of the prepared silica from GcHA

The comparison of elemental compositions of the guinea corn husk ash (GcHA) and the prepared silica as revealed by Energy Dispersive X-ray spectroscopy (EDX), (Figs. 1 and 2) with XRF results showed that the results were in close agreement. It is observed that silicon with the highest intensity is the major element present in the ash**.** This agrees with the XRF analysis result reported in Table 1. Consequence to this result, it is evident that silica can be prepared from GcHA. The guinea corn husk ash (GcHA) contains majorly silicon with little impurities of other elements. The intensity of the silicon (Si) in the prepared silica appears to be stronger (Fig. 2) than that of GcHA (Fig. 1). The disappearance of some minor elements like magnesium, potassium and calcium in the silica (Fig. 2) which were earlier present in GcHA (Fig. 1) suggested more purity of the silica. Therefore, purer silica has been extracted from the ash using sodium hydroxide (Shaik and Shaik, 2013). However, the presence of carbon and sulphur are likely to be contaminant during the analysis.

The FTIR spectroscopic study revealed the two major functional groups of silica, silanol (Si-OH) and siloxane (Si-O-Si) (Fig. 3). The summary of the observed functional groups is presented in Table 2. The broad absorption band at between 3433 cm^{-1} is assigned to –OH of silanol and absorbed water (Rani *et al*., 2014). The peaks at 954, 795 and 463 cm^{-1} correspond to asymmetry, symmetry and bending mode of SiO_2 respectively (Le *et al.*, 2013). The siloxane group of the silica is attributed to 1083 cm^{-1} (Chee and Yaacob, 2010). The surface functional group from FTIR analysis confirmed the material to be silica.

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Table.2: FTIR peaks of silica and the assigned bond

Assigned bond	Wave number (cm^{-1})
O-H	3433
Si-O-Si	1083
SiO_2 asymmetry mode	954
SiO_2 asymmetry mode	795
SiO_2 bending mode	463

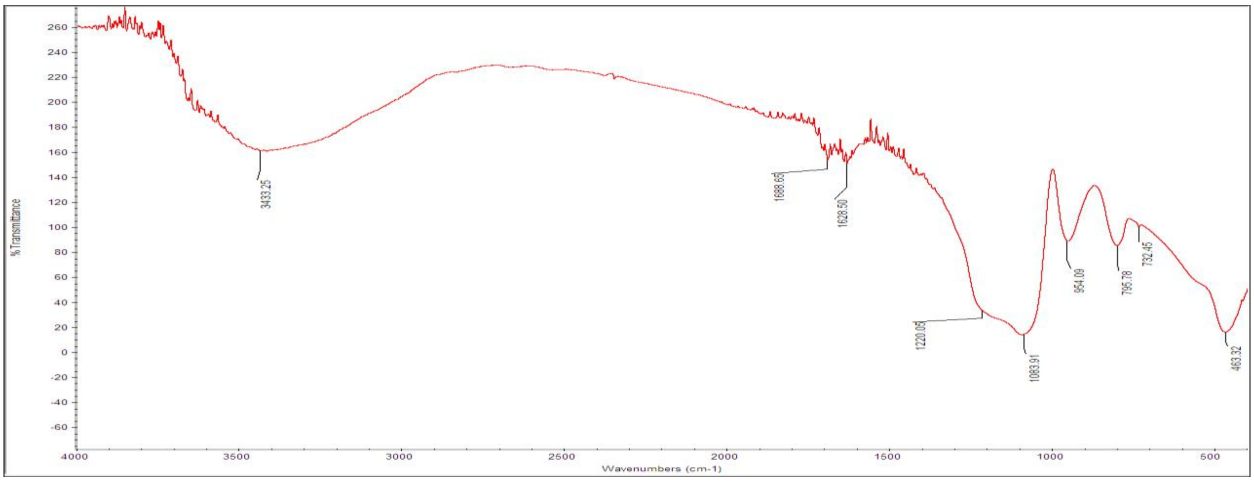

Fig. 3: FTIR spectrum of the prepared silica from GcHA

The morphology studied by Scanning Electron Microscopy (SEM) is shown in Figs. 4 and 5 for the GcHA and extracted silica respectively. The micrograph (Fig. 4) revealed flake like and irregular particles. These irregular particles of the ash morphology suggested different components of the ash. The Fig. 5 however shows agglomeration of regular and spherical shape particles but rough surface. This indicates assembly of similar particles of silicate ions as compare with the morphology of the ash which was irregular. Ndubuda and Nurudeen, (2015) reported the direct use of such ash in partial replacement of cement in concrete. It evident that higher quality and purity of silica can be extracted from ash contained silica by treatment with aqueous sodium hydroxide followed by precipitation using acid (Shaik and Shaik, 2013).

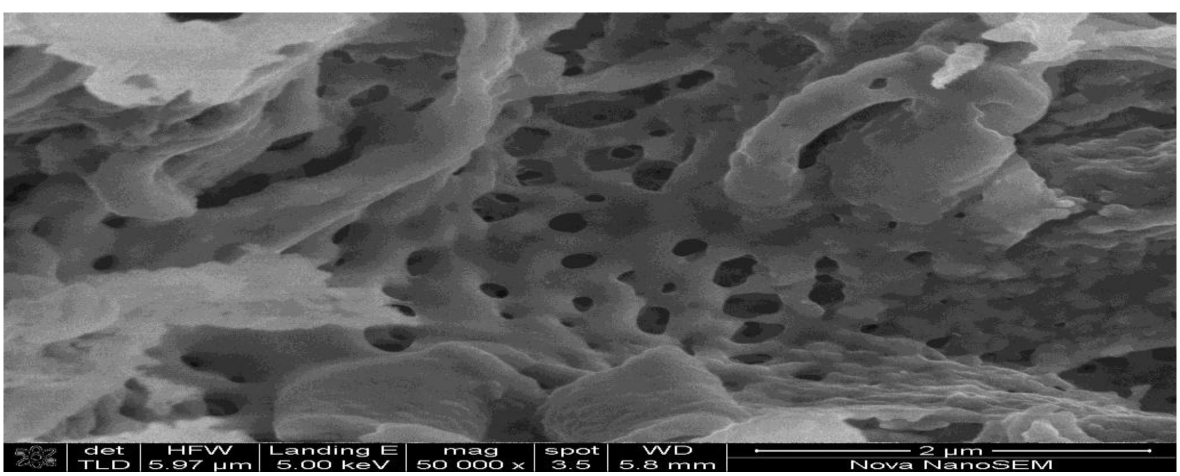

Fig.4: SEM micrograph of the GcHA

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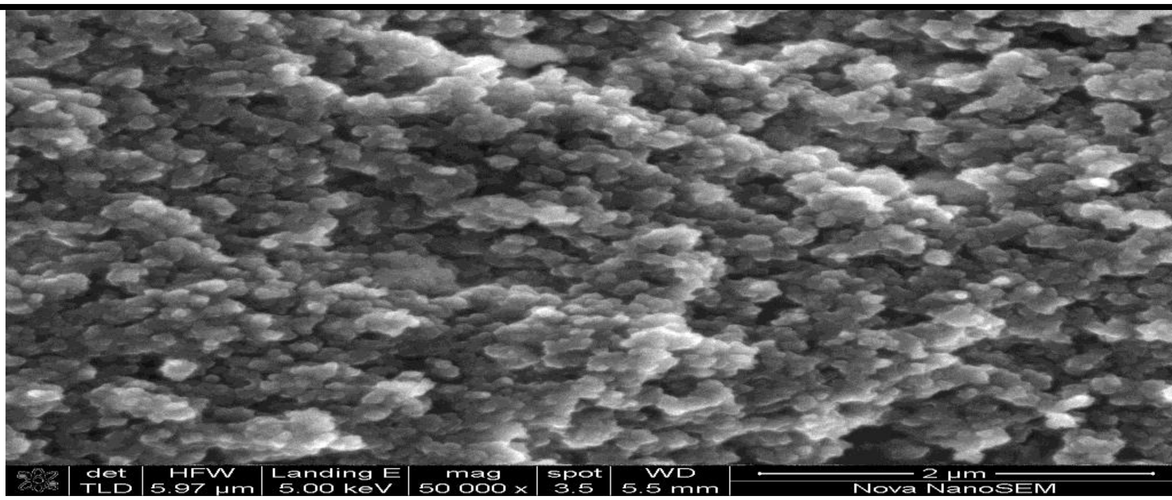

Fig.5: SEM micrograph of the prepared silica

The XRD pattern of the silica is presented in Fig. 6. From the figure, it can be said that the silica is an amorphous since the characteristic peak at 2θ cannot be seen (Le *et al*., 2013; Geetha *et al*., 2016).

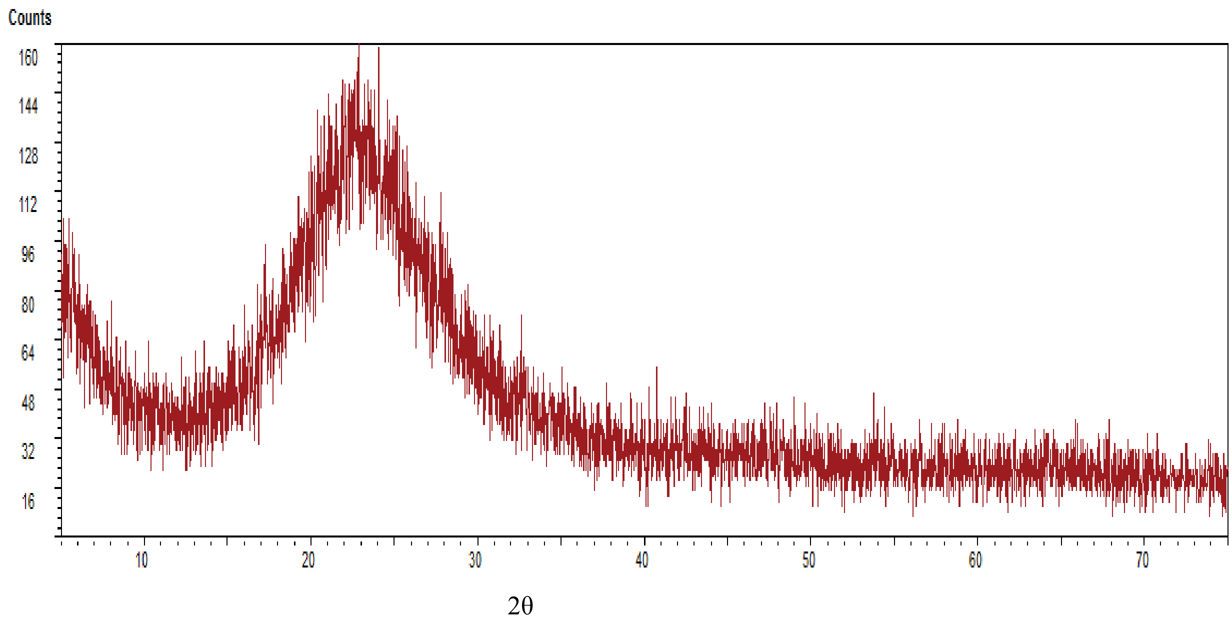

Fig. 6: XRD pattern of the prepared silica

IV. CONCLUSION

This study has successfully demonstrated the use of guinea corn husks, an agricultural waste for the preparation of silica. The characterization studies using FTIR and SEM-EDX confirmed the prepared material to be silica. Also XRD pattern indicated the amorphous form of silica. Therefore, it is found that amorphous silica, an important material which has numerous applications can be prepared in accordance with sustainable chemistry.

REFERENCES

[1] Akinloye S. A., Bankole G. M. and Medubi A. (2013) Effect of lime-Guinea Corn Husk Ash on the Engineering Properties of Lateritic Soil. Electronic Journal of Geotechnical Engineering, 19, 17693-17699

[2] Anyawu C. N., Ibeto C. N., Eze I. S. and Ezeoha S. L. (2013) Present and prospective energy use potentials of selected agricultural wastes in Nigeria. Journal of Renewable and Sustainable Energy, 5, 032703

[3] Chee C. Y. and Yaacob I. I. (2010) Influence of Nanosilica/Polyurethane Composite Coating on IR

Effectiveness and Visible Light Transmission Properties of Polyethylene. Advanced Materials Research, 97-101, 1669-1672

[4] Chongela J. (2015) Contribution of Agriculture Sector to Tanzanian Economy. American Journal of Research Communication, 3(7), 57-70

[5] Curran M. A. (2010) Biobased Materials. Kirk-Othmer Encyclopedia of Chemical Technology, ISBN: 9780471238966. John Wiley & Sons, Inc., Hoboken, NJ, 1-19.

[6] Edewor-Kuponiyi T. I. and Amuda O. S. (2013) Evaluation of the production of Oxalic Acid from some Solid Industrial Waste in Nigeria. International Journal of Basic and Applied Science, 2(1), 91-97

[7] Geetha D., Ananthiand A. and Ramesh P. S. (2016) Preparation and Characterization of Silica Material from Rice Husk Ash – An Economically Viable Method. Research & Reviews: Journal of Pure and Applied Physics, 4(3), 20-26

[8] Ghorbani F., Younesia H., Mehrabanb Z., Selikc M. S., Ghoreyshid A. A. and Anbiae M. (2013) Preparation and characterization of highly pure silica from sedge as agricultural waste and its utilization in the synthesis of mesoporous silica MCM-41. Journal of Taiwan Institute of Chemical Engineer, 44, 821–828

[9] Karagole Z. and Gur B. (2016) Sustainable Chemistry: Green Chemistry. Igdr University Journal of the Institute of Science and Technology, 6(2), 89-96

[10] Le V. H., Thuc C. N. H. and Thuc H. H., (2013) Synthesis of silica nanoparticles from Vietnamese rice husk by sol–gel method. Nanoscale Research Letters, 58(8): 1-10

[11] Nandanwar R., Singh P. and Haque F. Z. (2013) Synthesis and Properties of Silica Nanoparticles by Sol-gel Method for the Application in Green Chemistry. Material Science Research India, 10(1), 85-92

[12] Ndububa, E. and Nurudeen Y., (2015) Effect of Guinea Corn Husk Ash as Partial Replacement for Cement in Concrete, OSR Journal of Mechanical and Civil Engineering, 12(2), 40-45

[13] Ngumah C. C., Ogbulie J. N., Orji J. C., and Amadi E. S. (2013) Biogas Potential of Organic waste in Nigeria. Journal of Urban and Environmental Engineering, 7(1), 110-116

[14] Oyeleke S. B. and Jibrin N. M., (2009) Production of bioethanol from guinea cornhusk and millet husk. African Journal of Microbiology Research, 3(4), 147-152

[15] Rani K. M., Palanisamy P. N. and Sivakumar P., (2014) Synthesis and Characterization of Amorphous Nano-Silica from Biomass Ash. International Journal of Advanced Technology in Engineering and Science, 2(10), 71-76

[16] Simonyan K. J. and Fasina O. (2013) Biomass Resources and Bioenergy Potentials in Nigeria. African Journal of Agricultural Research, 8(40), 4975-4989

[17] Sivasubramanian S. and Sravanthi K., (2015) Synthesis and Characterization of Silica Nano Particles from Coconut Shell. International Journal of Pharma and Bio Sciences, 6(1), 530 – 536

[18] Shaikh I. R. and Shaikh A. A., (2013) Utilization of Wheat Husk Ash as Silica Source for the Synthesis of MCM-41 Type Mesoporous Silicates: A Sustainable Approach towards Valorization of the Agricultural Waste Stream, Research Journal of Chemical Sciences, 3(11), 66-72

[19] Sun L. and Gong K. (2001) Silicon-Based Materials from Rice Husks and Their Applications. Ind. Eng. Chem. Res., 40, 5861-5877

[20] Verma R. S. (2014) Greener and Sustainable Chemistry. Applied Sciences, 4, 493-497

[21] Wang W., Martin J. C., Fan X., Han A., Luo Z. and Luyi Sun L. (2012) Silica Nanoparticles and Frameworks from Rice Husk Biomass. ACS Applied Materials and Interfaces, 4 (2), 977–981